

## PATENT COOPERATION TREATY

PCT

NOTIFICATION OF THE RECORDING  
OF A CHANGE(PCT Rule 92bis.1 and  
Administrative Instructions, Section 422)

From the INTERNATIONAL BUREAU

To:

AWAPATENT AB  
P.O. Box 5117  
S-200 17 Malmö  
SUÈDE

Date of mailing (day/month/year) 27 June 2000 (27.06.00)	<b>IMPORTANT NOTIFICATION</b>
Applicant's or agent's file reference 2000263	
International application No. PCT/SE99/02057	International filing date (day/month/year) 12 November 1999 (12.11.99)

## 1. The following indications appeared on record concerning:

☒ the applicant
 ☐ the inventor
 ☐ the agent
 ☐ the common representative

## Name and Address

BOREALIS A/S  
Lyngby Hovedgade 96  
DK-2800 Lyngby  
Denmark

## State of Nationality

DK

## State of Residence

DK

Telephone No.

Facsimile No.

Teleprinter No.

## 2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

☒ the person
 ☒ the name
 ☒ the address
 ☒ the nationality
 ☒ the residence

## Name and Address

BOREALIS TECHNOLOGY OY  
P.O. Box 330  
FIN-06101 Porvoo  
Finland

## State of Nationality

FI

## State of Residence

FI

Telephone No.

Facsimile No.

Teleprinter No.

## 3. Further observations, if necessary:

## 4. A copy of this notification has been sent to:

<input checked="" type="checkbox"/> the receiving Office	<input checked="" type="checkbox"/> the designated Offices concerned
<input type="checkbox"/> the International Searching Authority	<input type="checkbox"/> the elected Offices concerned
<input type="checkbox"/> the International Preliminary Examining Authority	<input type="checkbox"/> other:

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer

Beate Giffo-Schmitt

Telephone No.: (41-22) 338.83.38

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents  
United States Patent and Trademark  
Office  
Box PCT  
Washington, D.C.20231  
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

<b>Date of mailing (day/month/year)</b> 21 August 2000 (21.08.00)	
<b>International application No.</b> PCT/SE99/02057	<b>Applicant's or agent's file reference</b> 2000263
<b>International filing date (day/month/year)</b> 12 November 1999 (12.11.99)	<b>Priority date (day/month/year)</b> 15 December 1998 (15.12.98)
<b>Applicant</b> DAMMERT, Ruth et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:  
11 May 2000 (11.05.00)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was  
☐ was not

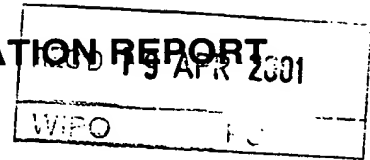
made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland  Facsimile No.: (41-22) 740.14.35	Authorized officer  Charlotte ENGER  Telephone No.: (41-22) 338.83.38
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## PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)



Applicant's or agent's file reference PC-2000263	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/SE99/02057	International filing date (day/month/year) 12/11/1999	Priority date (day/month/year) 15/12/1998
International Patent Classification (IPC) or national classification and IPC H01B3/44		
Applicant BOREALIS TECHNOLOGY OY et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 5 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand  11/05/2000	Date of completion of this report  11.04.2001
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0. Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  Komenda, C  Telephone No. +49 89 2399 8308  

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/SE99/02057

## I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

**Description, pages:**

1-22 as originally filed

**Claims, No.:**

1-13 with telefax of 08/03/2001

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/SE99/02057

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes:	Claims	1-13
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-13
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-13
	No:	Claims	

2. Citations and explanations  
**see separate sheet**

**Concerning paragraph V:**

1. Reference is made to the following documents:

D1: US-A-4983675

D2: WO-A1-9814516

D3: WO-A1-9517463

**2. Novelty (Art. 33(2) PCT):**

- 2.1** Document D1 discloses a composition comprising a crosslinkable polymer containing hydrolysable silane groups. The catalyst used to promote the crosslinking of the polymer is any conventional silanol condensation catalyst, different from the catalysts used according to the present application.
- 2.2** Document D2 discloses a semiconducting cross-linkable silane containing composition. The condensation catalysts used in D2 are, however, different from those used in the present application.
- 2.3** Document D3 discloses compositions comprising all the features of the claimed compositions. However, the process for the preparation of power cables and the power cables themselves are not disclosed in D1 (see D1, claims, p. 3, l. 25ff, examples).

The subject-matter of claims 1-13 is not disclosed in any of the cited documents and is, therefore, considered as being novel.

**3. Inventive step (Art 33(3) PCT):**

The subject-matter of claims 1 to 8 is directed to a specific medium or high voltage power cable having a specific sequence of semiconductor layers and insulating layers. Claims 9 to 13 are directed to a preparation process of the cables claimed in claims 1 to 8 using steam and superatmospheric pressure for crosslinking the polymer composition. Since none of the cited documents discloses the features of

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/SE99/02057

claims 1 to 13 nor renders them obvious, the subject-matter of claims 1 to 13 is regarded as involving an inventive step.

**4. Industrial applicability:**

The subject-matter of claims 1 to 13 is industrially applicable.

## CLAIMS

1. A medium to high voltage power cable comprising a conductor surrounded in order by an inner semi-conducting layer, an insulating layer, and an outer semi-conducting layer, characterised in that the insulating layer has a thickness of more than 2 mm and comprises the crosslinked product of a composition that comprises a crosslinkable polymer with hydrolysable silane groups, and a silanol condensation catalyst of formula I



or a precursor thereof, Ar being a benzene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical(s) is 8-20, or a naphthalene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical(s) is 4-18, and the catalyst of formula I containing 14-28 carbon atoms in total.

2. A medium to high voltage power cable as claimed in claim 1, wherein the insulating layer has a thickness of more than 5 mm.

3. A medium to high voltage power cable as claimed in claim 1 or 2, wherein the composition is hydrophilic.

4. A medium to high voltage power cable as claimed in claim 3, wherein the crosslinkable polymer has hydrophilic groups selected from siloxane, amide, anhydride, carboxylic, carbonyl, hydroxyl, and ester groups.

5. A medium to high voltage power cable as claimed in any one of the preceding claims, wherein the crystalline part of the polymer is at most 60% by weight.

6. A medium to high voltage power cable as claimed in any one of the preceding claims, wherein the



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hydrocarbyl radical in formula I is an alkyl substituent with 10-18 carbon atoms.

7. A medium to high voltage power cable as claimed in claim 6, wherein the alkyl substituent has 12 carbon atoms and is selected from dodecyl and tetrapropyl.

8. A composition as claimed in any one of the preceding claims, wherein the polymer composition includes 0.0001-3% by weight of silanol condensation catalyst.

9. A process of preparing a medium to high voltage power cable according to any one of claims 1-8, in which a conductor is surrounded in order by an inner semi-conducting layer, an insulating layer comprising a crosslinkable polymer with hydrolysable silane groups, and an outer semi-conducting layer to form a cable, characterised in that the cable is crosslinked in the presence of steam at a superatmospheric pressure.

10. A process according to claim 9, wherein the crosslinking is carried out in a vulcanising tube.

11. A process according to claim 9 or 10, wherein the crosslinking is carried out at a pressure of 0.2-2.5 MPa.

12. A process according to claim 11, wherein the crosslinking is carried out at a pressure of 0.8-1.2 MPa.

13. A process according to any one of claims 9-12, wherein the crosslinking is carried out in the presence of saturated steam.

# PCT REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference  
(if desired)(12 characters maximum) 2000263

## Box No. I TITLE OF INVENTION

A POWER CABLE INSULATION LAYER, A PROCESS FOR THE PREPARATION THEREOF, AND A COMPOSITION THEREFOR

## Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

BOREALIS A/S  
Lyngby Hovedgade 96  
DK-2800 LYNGBY  
DENMARK

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State (that is, country) of nationality: DENMARK

State (that is, country) of residence: DENMARK

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

## Box No. III FURTHER APPLICANT(S) AND/OR /FURTHER INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

DAMMERT, Ruth  
Smyckegatan 50  
S-426 50 V. FRÖLUNDA  
SWEDEN

This person is:

☐ applicant only  
☒ applicant and inventor  
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality: SWEDEN

State (that is, country) of residence: SWEDEN

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☒ Further applicants and/or (further) inventors are indicated on a continuation sheet

## Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

AWAPATENT AB  
Box 5117  
SE-200 71 MALMÖ  
SWEDEN

Telephone No.

+46 40 98 51 00

Facsimile No.

+46 40 26 05 16

Teleprinter No.

☐ Address for correspondence: Mark this check-box where no agent or common representative has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent

Form PCT/RO/101 (continuation sheet) (July 1998; reprint July 1999)

*See Notes to the request form*

**Box No. V DESIGNATION OF STATES**

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

**Regional Patent**

- ☒ **AP** **ARIPO Patent:** GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SL Sierra Leone, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ **EA** **Eurasian Patent:** AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ **EP** **European Patent:** AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ **OA** **OAPI Patent:** BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line)

**National Patent (if other kind of protection or treatment desired, specify on dotted line):**

- |   |  |  |
|---|--|--|
| <input checked="" type="checkbox"/> <b>AE</b> United Arab Emirates                  | <input checked="" type="checkbox"/> <b>LR</b> Liberia  |  |
| <input checked="" type="checkbox"/> <b>AL</b> Albania                               | <input checked="" type="checkbox"/> <b>LS</b> Lesotho  |  |
| <input checked="" type="checkbox"/> <b>AM</b> Armenia                               | <input checked="" type="checkbox"/> <b>LT</b> Lithuania  |  |
| <input checked="" type="checkbox"/> <b>AT</b> Austria +Utility Model                | <input checked="" type="checkbox"/> <b>LU</b> Luxembourg   |  |
| <input checked="" type="checkbox"/> <b>AU</b> Australia                             | <input checked="" type="checkbox"/> <b>LV</b> Latvia   |  |
| <input checked="" type="checkbox"/> <b>AZ</b> Azerbaijan                            | <input checked="" type="checkbox"/> <b>MD</b> Republic of Moldova  |  |
| <input checked="" type="checkbox"/> <b>BA</b> Bosnia and Herzegovina                | <input checked="" type="checkbox"/> <b>MG</b> Madagascar   |  |
| <input checked="" type="checkbox"/> <b>BB</b> Barbados                              | <input checked="" type="checkbox"/> <b>MK</b> The former Yugoslav Republic of Macedonia                      |  |
| <input checked="" type="checkbox"/> <b>BG</b> Bulgaria                              |  |  |
| <input checked="" type="checkbox"/> <b>BR</b> Brazil                                | <input checked="" type="checkbox"/> <b>MN</b> Mongolia   |  |
| <input checked="" type="checkbox"/> <b>BY</b> Belarus                               | <input checked="" type="checkbox"/> <b>MW</b> Malawi   |  |
| <input checked="" type="checkbox"/> <b>CA</b> Canada                                | <input checked="" type="checkbox"/> <b>MX</b> Mexico   |  |
| <input checked="" type="checkbox"/> <b>CH and LI</b> Switzerland and Liechtenstein  | <input checked="" type="checkbox"/> <b>NO</b> Norway   |  |
| <input checked="" type="checkbox"/> <b>CN</b> China                                 | <input checked="" type="checkbox"/> <b>NZ</b> New Zealand  |  |
| <input checked="" type="checkbox"/> <b>CU</b> Cuba                                  | <input checked="" type="checkbox"/> <b>PL</b> Poland   |  |
| <input checked="" type="checkbox"/> <b>CZ</b> Czech Republic +Utility Model         | <input checked="" type="checkbox"/> <b>PT</b> Portugal   |  |
| <input checked="" type="checkbox"/> <b>DE</b> Germany +Utility Model                | <input checked="" type="checkbox"/> <b>RO</b> Romania  |  |
| <input checked="" type="checkbox"/> <b>DK</b> Denmark +Utility Model                | <input checked="" type="checkbox"/> <b>RU</b> Russian Federation   |  |
| <input checked="" type="checkbox"/> <b>EE</b> Estonia +Utility Model                | <input checked="" type="checkbox"/> <b>SD</b> Sudan  |  |
| <input checked="" type="checkbox"/> <b>ES</b> Spain                                 | <input checked="" type="checkbox"/> <b>SE</b> Sweden   |  |
| <input checked="" type="checkbox"/> <b>FI</b> Finland +Utility Model                | <input checked="" type="checkbox"/> <b>SG</b> Singapore  |  |
| <input checked="" type="checkbox"/> <b>GB</b> United Kingdom                        | <input checked="" type="checkbox"/> <b>SI</b> Slovenia   |  |
| <input checked="" type="checkbox"/> <b>GD</b> Grenada                               | <input checked="" type="checkbox"/> <b>SK</b> Slovakia +Utility Model  |  |
| <input checked="" type="checkbox"/> <b>GE</b> Georgia                               | <input checked="" type="checkbox"/> <b>SL</b> Sierra Leone   |  |
| <input checked="" type="checkbox"/> <b>GH</b> Ghana                                 | <input checked="" type="checkbox"/> <b>TJ</b> Tajikistan   |  |
| <input checked="" type="checkbox"/> <b>GM</b> Gambia                                | <input checked="" type="checkbox"/> <b>TM</b> Turkmenistan   |  |
| <input checked="" type="checkbox"/> <b>HR</b> Croatia                               | <input checked="" type="checkbox"/> <b>TR</b> Turkey   |  |
| <input checked="" type="checkbox"/> <b>HU</b> Hungary                               | <input checked="" type="checkbox"/> <b>TT</b> Trinidad and Tobago  |  |
| <input checked="" type="checkbox"/> <b>ID</b> Indonesia                             | <input checked="" type="checkbox"/> <b>UA</b> Ukraine  |  |
| <input checked="" type="checkbox"/> <b>IL</b> Israel                                | <input checked="" type="checkbox"/> <b>UG</b> Uganda   |  |
| <input checked="" type="checkbox"/> <b>IN</b> India                                 | <input checked="" type="checkbox"/> <b>US</b> United States of America                                       |  |
| <input checked="" type="checkbox"/> <b>IS</b> Iceland                               |  |  |
| <input checked="" type="checkbox"/> <b>JP</b> Japan                                 | <input checked="" type="checkbox"/> <b>UZ</b> Uzbekistan   |  |
| <input checked="" type="checkbox"/> <b>KE</b> Kenya                                 | <input checked="" type="checkbox"/> <b>VN</b> Viet Nam   |  |
| <input checked="" type="checkbox"/> <b>KG</b> Kyrgyzstan                            | <input checked="" type="checkbox"/> <b>YU</b> Yugoslavia   |  |
| <input checked="" type="checkbox"/> <b>KP</b> Democratic People's Republic of Korea | <input checked="" type="checkbox"/> <b>ZA</b> South Africa   |  |
|   | <input checked="" type="checkbox"/> <b>ZW</b> Zimbabwe   |  |
| <input checked="" type="checkbox"/> <b>KR</b> Republic of Korea                     | Check-boxes reserved for designating States which have become party to the PCT after issuance of this sheet: |  |
| <input checked="" type="checkbox"/> <b>KZ</b> Kazakhstan                            |  |  |
| <input checked="" type="checkbox"/> <b>LC</b> Saint Lucia                           | <input checked="" type="checkbox"/> <b>CR</b> Costa Rica   | <input checked="" type="checkbox"/> <b>TZ</b> Tanzania |
| <input checked="" type="checkbox"/> <b>LK</b> Sri Lanka                             | <input checked="" type="checkbox"/> <b>DM</b> Dominica   | <input checked="" type="checkbox"/> <b>MA</b> Morocco  |

**Precautionary Designation Statement:** In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)

Box No. VI PRIORITY CLAIM <input type="checkbox"/> Further priority claims are indicated in the Supplement Box.				
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application:* regional Office	international application: receiving Office
item (1) 15 December 1998 15.12.1998	9804323-5	SWEDEN		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s): \_\_\_\_\_

\* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.

### Box No. VII INTERNATIONAL SEARCHING AUTHORITY

<b>Choice of International Searching Authority (ISA)</b> (If two or more International Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):	<b>Request to use results of earlier search; reference to that search</b> (if an earlier search has been carried out by or requested from the International Searching Authority):		
ISA / SE	Date (day/month/year)	Number	Country (or regional Office)
	15.12.1998	SE 98/01428	SWEDEN

### Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following number of sheets: request : 4 description (excluding sequence listing part) : 22 claims : 2 abstract : 1 drawings : sequence listing part of description : Total number of sheets : 29	This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input checked="" type="checkbox"/> separate signed power of attorney 3. <input type="checkbox"/> copy of general power of attorney; reference No., if any: 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): 6. <input type="checkbox"/> translation of international applications into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input checked="" type="checkbox"/> other (specify): Subauthorisation. Copy of ITS-report. Official Letter.
Figure of the drawings which should accompany the abstract:	Language of filing of the international application: English

### Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

11 November 1999



Erik Wiklund

Authorised Agent

For receiving Office use only		2. Drawings:  <input type="checkbox"/> received:  <input type="checkbox"/> not received:
1. Date of actual receipt of the Purported international application:		
3. Corrected date of actual receipt due to later but Timely received papers or drawings completing the purported international application:		
4. Date of timely receipt of the required Corrections under PCT Article 11(2):		
5. International Searching Authority (if two or more are competent): ISA	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.	

For International Bureau use only

Date of receipt of the record copy by the International Bureau:

# PATENT COOPERATION TREATY

PCT

## NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

From the INTERNATIONAL BUREAU

To:

AWAPATENT AB  
P.O. Box 5117  
S-200 17 Malmö  
SUÈDE

Date of mailing (day/month/year) 22 June 2000 (22.06.00)		<div style="border: 2px solid black; padding: 5px; transform: rotate(-2deg);"> <b>RECEIVED</b>  2000-06-30  AWAPATENT, Malmö </div>	
Applicant's or agent's file reference 2000263			
International application No. PCT/SE99/02057		International filing date (day/month/year) 12 November 1999 (12.11.99)	Priority date (day/month/year) 15 December 1998 (15.12.98)
Applicant BOREALIS A/S et al			

### IMPORTANT NOTICE

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:  
AU,CN,JP,KP,KR,US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE,AL,AM,AP,AT,AZ,BA,BB,BG,BR,BY,CA,CH,CR,CU,CZ,DE,DK,DM,EA,EE,EP,ES,FI,GB,GD,GE,  
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<b>(21) International Application Number:</b> PCT/SE99/02057 <b>(22) International Filing Date:</b> 12 November 1999 (12.11.99) <b>(30) Priority Data:</b> 9804323-5 15 December 1998 (15.12.98) SE <b>(71) Applicant (for all designated States except US):</b> BOREALIS A/S [DK/DK]; Lyngby Hovedgade 96, DK-2800 Lyngby (DK). <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> DAMMERT, Ruth [SE/SE]; Smyckegatan 50, S-426 50 V. Frölunda (SE). SULTAN, Bernt-Åke [SE/SE]; Söbackevägen 16, S-444 42 Stenungsund (SE). FAGRELL, Ola [SE/SE]; Gulkragevägen 4, S-444 45 Stenungsund (SE). <b>(74) Agent:</b> AWAPATENT AB; P.O. Box 5117, S-200 17 Malmö (SE).			<b>(81) Designated States:</b> AE, AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), DM, EE, EE (Utility model), ES, FI, FI (Utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, L'I, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> With international search report.
<b>(54) Title:</b> A POWER CABLE INSULATION LAYER, A PROCESS FOR THE PREPARATION THEREOF, AND A COMPOSITION THEREFOR			
<b>(57) Abstract</b>  A composition for an insulating layer of a medium to high voltage power cable is disclosed. The composition comprises a cross-linkable polymer with hydrolysable silane groups, and a silanol condensation catalyst of formula (I) $\text{ArSO}_3\text{H}$ or a precursor thereof, Ar being a benzene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical (s) is 8-20, or a naphthalene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical(s) is 4-18, and the catalyst of formula (I) containing 14-28 carbon atoms in total. A medium to high voltage cable with an insulating layer that has a thickness of more than 2 mm and that comprises the cross-linked product of the above mentioned composition is also disclosed, as is a process of preparing such a cable by cross-linking the cable in the presence of steam at a superatmospheric pressure. Preferably the crosslinking is carried out with saturated steam in a vulcanising tube at about 0.8-1.2 MPa and 170-190°C.			

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A POWER CABLE INSULATION LAYER, A PROCESS FOR THE  
PREPARATION THEREOF, AND A COMPOSITION THEREFOR

Field of the invention

The present invention relates to a composition for an insulating layer of an electric power cable, to an electric power cable comprising an insulating layer of said composition, and to a process for the preparation of an insulating layer comprising the crosslinked composition.

Technical background

Electric power cables for medium voltages (MV; 6-68 kV) and high voltages (HV; >68 kV) are composed of a plurality of polymer layers extruded around an electric conductor. The electric conductor is usually coated first with an inner semiconductor layer followed by an insulating layer, then an outer semiconductor layer followed by water barrier layers, if any, and on the outside a sheath layer. The layers of the cable are based on different types of ethylene polymers which usually are crosslinked.

Crosslinked ethylene polymers are used for the insulating layer. LDPE (low-density polyethylene, i.e. polyethylene prepared by radical polymerisation at a high pressure) is today the predominant cable insulating material.

In addition to the polyethylene base resin insulating compositions usually contain additives to improve the physical properties of the insulating layer of the electric cable and to increase its resistance to the influence of different conditions. The total amount of the additives is generally about 0.3-5% by weight, preferably about 1-4% by weight. The additives include stabilising additives such as antioxidants to counteract decomposition due to oxidation, radiation, etc.; lubricating additives, such as stearic acid; additives for water-tree

resistance, such as polyethylene glycol, silicones etc.; and crosslinking additives such as peroxides to aid in the crosslinking of the ethylene polymer of the insulating composition.

A common way to crosslink the insulating layer of a power cable is by peroxide crosslinking, i.e. to add a peroxide crosslinking agent to the insulating composition, shape the composition into a cable insulating layer, e.g. by extrusion and crosslink the insulating layer by heating to decompose the peroxide crosslinking agent. Normally about 1 to 3% by weight, preferably about 2% by weight of peroxide crosslinking agent is added to the composition.

The crosslinking is performed after the extrusion of the insulating layer in a long, e.g. about 100-200 m, multi-zone vulcanisation tube where crosslinking should take place as rapidly and completely as possible. The vulcanising tube, which preferably is of a continuous vulcanising (CV) type, has at least one heating zone and at least one cooling zone and the vulcanising is initiated by the heat emitted in one or more of the heating zones of the vulcanising tube. Generally, the temperature in the heating zone(s) of the vulcanising tube is up to about 400°C. A nitrogen-gas pressure is also applied in the tube, and contributes to prevent oxidation processes by keeping away oxygen of the air and to reduce the formation of microcavities, so-called voids, in the polymer layers by reducing the expansion of the gases resulting from the decomposition of the radical-forming crosslinking agent.

The peroxide crosslinking agents used for the crosslinking of insulating layers of electric power cables as described above present some problems, both from a technical and an environmental point of view. When they decompose, gaseous decomposition products are formed and these products cause formation of microvoids in the cable insulation, voids from which water trees may later arise

during ageing of the cables. Furthermore the decomposition products may have obnoxious smell and may even cause allergies in sensitive persons.

In view of the disadvantages of the peroxide cross-linking agents it would be a technical progress if cross-linking could be achieved by way of other means without the disadvantages of the peroxide crosslinking agents.

It is also known to crosslink ethylene polymers by way of crosslinkable groups such as hydrolysable silane groups in the polymer. The crosslinking of polymers with hydrolysable silane groups is carried out by so-called moisture curing. In a first step, the silane groups are hydrolysed under the influence of water or steam, resulting in the splitting-off of alcohol and the formation of silanol groups. In a second step, the silanol groups are crosslinked by a condensation reaction splitting off water. In both steps, a so-called silanol condensation catalyst is used as a catalyst.

Silanol condensation catalysts include carboxylates of metals, such as tin, zinc, iron, lead and cobalt; organic bases; inorganic acids; and organic acids. In practice dibutyl tin dilaurate (DBTL) is generally used as the silanol condensation catalyst.

However, silanol condensation catalysts such as DBTL give poor performance at normal ambient temperatures and relative humidities. To function satisfactorily they require a sauna or water bath at an elevated temperature in the order of 70-100°C. Although the crosslinking is speeded up by increasing the temperature, it may take 6-24 hours to carry out the crosslinking for a thin LV cable and from 1 to 7 days for an MV cable, depending on thickness and curing temperature. Moreover, DBTL is a tin-organic catalyst which is negative from an environmental point of view. A particular problem is encountered in connection with crosslinking of insulating layers of medium voltage and high voltage power cables because of the difficulty for the water to reach and completely

crosslink the layer. This is due to the fact that the insulating layer is covered by the outer semi-conductor layer and that the insulating layer has a substantial thickness. Generally, the insulating layer of a medium voltage power cable is at least about 2 mm thick, typically at least 2.3 mm thick and the thickness increases with the voltage of the cable. Thus, the insulating layer of a 10 kV cable typically has a thickness of about 3.6 mm and in a 20 kV cable this layer typically has a thickness of about 5.5 mm. The more layers that cover the insulating layer and the greater its thickness the more difficult it will be and the longer it will take for the water to reach and crosslink the insulating layer. Indeed, polymers containing hydrolysable silane groups have hitherto not been considered feasible as insulating layers for power cables of voltages over 20 kV, i.e. having an insulating layer thickness of more than about 5 mm, due to the excessive time needed for crosslinking.

From the above it is evident that the crosslinking of electric power cables using hydrolysable silane group containing polymers for the insulating layer is also associated with several disadvantages and that it would mean an important technical progress if crosslinking could be achieved without these disadvantages.

#### Summary of the invention

It is an object of the present invention to provide a moisture curable insulating layer composition for a medium to high voltage power cable that alleviates or eliminates the above mentioned problems of the prior art.

It is another object of the present invention to provide a medium to high voltage power cable comprising a crosslinked insulating layer of the above mentioned insulating layer composition.

It is still another object of the present invention to provide a process for preparing such a medium to high voltage power cable.

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According to one aspect of the present invention it has been found that it is possible to use a crosslinkable hydrolysable silane group containing polymer as a base resin for the insulating layer of a medium to high voltage power cable if the prior art silanol condensation catalyst, such as DBTL is replaced by a particular sulphonic acid catalyst.

According to another aspect of the present invention it has been found that the crosslinking of the insulating layer may be further enhanced, particularly in connection with thick insulating layers, if when using said sulphonic acid as a silanol condensation catalyst the insulating layer composition is made more hydrophilic and/or more amorphous.

According to still another aspect of the present invention it has been found that the medium to high voltage power cable may be prepared by carrying out the crosslinking of the cable at superatmospheric pressure in the presence of steam, preferably in a vulcanising tube usually used for the crosslinking of peroxide crosslinkable power cables.

The present invention thus provides a composition for an insulating layer of a medium to high voltage power cable, characterised in that the composition comprises a crosslinkable polymer with hydrolysable silane groups, and a silanol condensation catalyst of formula I



or a precursor thereof, Ar being a benzene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical(s) is 8-20, or a naphthalene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical(s) is 4-18, and the catalyst of formula I containing 14-28 carbon atoms in total.

5 The present invention further provides a medium to  
high voltage power cable comprising a conductor surround-  
ed in order by an inner semi-conducting layer, an  
10 insulating layer, and an outer semi-conducting layer,  
5 characterised in that the insulating layer has a thick-  
ness of more than 2 mm and comprises the crosslinked  
product of a composition as defined above.

15 Still further, the present invention provides a  
process of preparing a medium to high voltage power  
10 cable as defined above, characterised in that the cable  
is crosslinked in the presence of steam at a super-  
atmospheric pressure.

20 Further characterising features and advantages of  
the present invention will appear from the following  
15 description and the appended claims.

25 Detailed description of the invention

A silanol condensation catalyst of the above defined  
type is disclosed in WO 95/17463 for the crosslinking of  
polymers with hydrolysable silane groups in general. How-  
30 ever, it does not disclose the specific use of such a  
catalyst for the crosslinking of insulating layers of  
medium to high voltage power cables or that it may sur-  
prisingly overcome the particular problems associated  
35 with the crosslinking of such insulating layers. Further,  
25 as will be explained and illustrated in more detail  
below, this catalyst quite surprisingly gives improved  
water treeing characteristics to the insulating layer of  
a medium to high voltage power cable.

40 According to a particularly preferred aspect of the  
30 invention the composition of the insulating material is  
changed in a way that facilitates the migration of water  
into it. This may be achieved by incorporating comonomers  
45 containing hydrophilic functions into the matrix resin,  
by blending in water absorbing components, e.g. via a  
35 master batch, into the composition or by decreasing the  
crystallinity of the matrix resin. The last alternative  
50 will leave a more amorphous structure with more room for

5 small molecules like water to migrate into it. Of course,  
if polar comonomers are introduced into the matrix resin  
they will also decrease the crystallinity and thus have a  
10 double effect. The crystalline part of the polymer matrix  
5 thus should be at the most about 60% by weight.

As examples of preferred hydrophilic functions or  
groups that may be introduced into the polymer (matrix  
15 resin) the following may be mentioned: siloxane, amide,  
anhydride, carboxylic, carbonyl, hydroxyl, and ester  
10 groups.

With regard to the silanol condensation catalyst of  
20 formula I it is preferred that the hydrocarbyl radical in  
formula I is an alkyl substituent with 10-18 carbon  
atoms. More preferably the alkyl substituent has 12  
15 carbon atoms and is selected from dodecyl and tetra-  
propyl.  
25

It is further preferred that the polymer composition  
includes 0.0001-3% by weight of silanol condensation  
catalyst.

20 As indicated above, the present invention provides a  
composition that preferably is hydrophilic and/or amorph-  
ous and that comprises a polymer with hydrolysable silane  
groups and a silanol condensation catalyst having the  
30 formula I. In particular, the composition is contemplated  
35 for use as an insulating layer of a medium to high volt-  
age power cable. Other applications for the composition  
may, however, also be contemplated such as pipes, parti-  
40 cularly water pipes and gas pipes, and products made by  
injection or rotational moulding.

30 With regard to the medium to high voltage power  
cable according to the present invention it is preferred  
that the insulating layer has a thickness of more than 4  
45 mm, more preferably more than 5 mm. This corresponds  
approximately to a 10 kV cable and a 20 kV cable, respec-  
35 tively.

50 As mentioned above, the process according to the  
present invention for preparing a medium to high voltage

5 power cable comprising the composition of the invention  
as the insulating layer of the cable, is characterised in  
that the crosslinking is carried out in the presence of  
10 steam at a superatmospheric pressure. This is in contrast  
5 to conventional crosslinking of hydrolysable silane group  
containing polymers which is carried out at atmospheric  
pressure in the presence of moisture, i.e. steam or  
15 water. Preferably the crosslinking of the present inven-  
tion is carried out in the presence of saturated steam at  
10 the pressure in question. The pressure preferably lies in  
the range from about 0.2 MPa to about 2.5 MPa, more  
preferably from about 0.2 MPa to about 1.5 MPa, and most  
20 preferably from about 0.8 MPa to about 1.2 MPa. A  
pressure of 0.8 MPa corresponds to saturated steam of a  
15 temperature of about 170°C and 1.2 MPa corresponds to  
saturated steam of a temperature of about 190°C.

25 It is preferred, especially where the hydrophilicity  
is low and the crystallinity is relatively high, like  
when an ethylene/vinyl trimethoxysilane copolymer is used  
20 as the base resin, that the crosslinking is that the  
crosslinking is carried out in a vulcanising tube, such  
as a CV tube as described above normally used for the  
crosslinking of peroxide crosslinkable power cables.

35 In the following the crosslinkable hydrolysable  
25 silane group containing polymer used as the base resin of  
the insulating layer composition according to the present  
invention will be described.

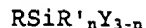
40 The crosslinkable base resin generally is an olefin  
copolymer or graft polymer which contains hydrolysable  
30 silane groups and which is crosslinked under the  
influence of water and at least one silanol condensation  
catalyst. Specifically, the crosslinkable polymer is an  
45 ethylene copolymer or homopolymer containing crosslink-  
able silane groups introduced either by copolymerisation  
35 or graft polymerisation.

50 Preferably, the silane-containing polymer has been  
obtained by copolymerisation of an olefin, suitably



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ethylene, and an unsaturated silane compound represented by the formula



wherein

R is an ethylenically unsaturated hydrocarbyl, hydrocarbyloxy or (meth)acryloxy hydrocarbyl group,

R' is an aliphatic saturated hydrocarbyl group,

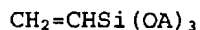
Y which may be same or different, is a hydrolysable organic group, and

n is 0, 1 or 2.

If there is more than one Y group, these do not have to be identical.

Special examples of the unsaturated silane compound are those wherein R<sub>1</sub> is vinyl, allyl, isopropenyl, butenyl, cyclohexenyl or gamma-(meth)acryloxy propyl; Y is methoxy, ethoxy, formyloxy, acetoxy, propionyloxy or an alkyl- or arylamino group; and R', if present, is a methyl, ethyl, propyl, decyl or phenyl group.

A preferred unsaturated silane compound is represented by formula



wherein A is a hydrocarbyl group having 1-8 carbon atoms, preferably 1-4 carbon atoms.

The most preferred compounds are vinyl trimethoxysilane, vinyl triethoxysilane, gamma-(meth)acryloxypropyltrimethoxysilane and vinyl triacetoxysilane or combinations of two or more thereof.

The copolymerisation of the olefin (ethylene) and the unsaturated silane compound may be carried out under any suitable conditions resulting in the copolymerisation of the two monomers.

The silane-containing polymer according to the invention suitably contains 0.001-15% by weight of the

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silane compound, preferably 0.01-5% by weight, most preferably 0.1-3% by weight.

Moreover, the copolymerisation (or grafting) may be implemented in the presence of one or more other comonomers which can be copolymerised (or grafted) with the two monomers. The copolymerisation (or grafting) with other comonomers besides the unsaturated silane comonomer is applied in particular when it is desired to make the crosslinkable polymer composition hydrophilic and/or amorphous. In that case the comonomer (or termonomer) should include at least one hydrophilic group, such as an acyl group, an hydroxyl group or an ester group. As non-limiting examples of hydrophilic comonomers may be mentioned the following: (a) vinyl carboxylate esters, such as vinyl acetate and vinyl pivalate, (b) (meth)acrylates, such as methyl(meth)acrylate, ethyl(meth)acrylate and butyl(meth)acrylate, (c) olefinically unsaturated carboxylic acids, such as (meth)acrylic acid, maleic acid and fumaric acid, (d) (meth)acrylic acid derivatives, such as (meth)acrylonitrile and (meth)acrylic amide, and (e) vinyl ethers, such as vinyl methyl ether and vinyl phenyl ether. Amongst these comonomers, vinyl esters of monocarboxylic acids having 1-4 carbon atoms, such as vinyl acetate, and (meth)acrylates of alcohols having 1-4 carbon atoms, such as methyl(meth)acrylate, are preferred. Especially preferred comonomers are butyl acrylate, ethyl acrylate and methyl acrylate. Two or more such olefinically unsaturated compounds may be used in combination. The term '(meth)acrylic acid' is intended to embrace both acrylic acid and methacrylic acid. The hydrophilic comonomer contents of the hydrophilic copolymer may amount to as much 40% by weight of the copolymer but more normally amounts to between 3 and 20% by weight. The amount will to a great deal depend on the flexibility wanted in the resin, since the higher the comonomer content the more flexible and rubberlike will be the cable insulation.

If using a graft polymer, this may be produced e.g. by the methods described in US 3,646,155 and US 4,117,195.

Because of the increased hydrophilicity of the composition according to the above aspect of the present invention care should be taken to keep the composition out of contact with water before shaping the composition into a cable insulating layer in order to avoid undesired premature crosslinking or scorching of the composition. Alternatively or in addition a so-called scorch retarder may be added to the composition. Such scorch retarders are known per se and comprise compounds that react chemically with water such as e.g. hydrolysable silane compounds.

The silanol condensation catalyst defined above and used at the present invention is distinguished by being a benzene or naphthalene sulphonic acid that is sufficiently lipophilic to be compatible with the polymer composition to be crosslinked. To achieve such lipophilicity, the hydrocarbon group of the alkylaryl sulphonic acid must have a certain size and must, e.g. in the case where the acid is a benzene sulphonic acid, have an alkyl substituent containing at least 8 carbon atoms, and in the case where the acid is a naphthalene sulphonic acid the alkyl substituent must contain at least 4 carbon atoms. If the alkyl group does not have such a size that the lipophilicity requirement is met, the catalyst is not compatible with the polymer composition but will be released therefrom upon crosslinking in aqueous solution, thus impairing crosslinking efficiency.

Due to commercial availability, it is preferred that the aryl group is a benzene ring, substituted with an alkyl substituent containing 12 carbon atoms.

The currently most preferred compounds of formula I are dodecyl benzene sulphonic acid and tetrapropyl benzene sulphonic acid.

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power cables where mainly peroxide crosslinking has been used earlier. While theoretically an alternative to peroxide crosslinking, crosslinking of hydrolysable silane group containing polymers with conventional silanol condensation catalysts has found little or no practical use. The reason for this is the disadvantages involved such as very long crosslinking times that reduce the productivity. Moreover, it has not been considered economically feasible to crosslink silane containing insulating layers in power cables of more than 20 kV due to the thickness of the insulating layer (5 mm or more) and the difficulty of moisture curing such thick layers.

Compared to the prior art insulating compositions the insulating composition according to the present invention represents an important progress due to the substantial advantages it presents.

Thus, due to the absence of decomposable peroxide crosslinking agent the silane group containing polymer composition of the present invention may be extruded at higher melt temperatures, such as about 180-200°C, than conventional peroxide crosslinkable compositions. Without scorching these higher melt temperatures result in a higher output and a higher line speed.

Further, due to the absence of peroxide crosslinking agent no environmental pollutants from peroxide decomposition are generated. The particular silanol condensation catalyst used according to the present invention is no environmental pollutant; it is not dissolved from the polymer composition in water baths; and it does not discolour the polymer composition.

Compared to peroxide crosslinked insulating polymer compositions which require separate processing steps for the compounding of additives and soaking of the peroxide crosslinking agent, the present invention requires none of these steps and therefore results in a more simple overall process with fewer processing steps.

Thanks to fewer processing steps there is less risk of contaminating the composition which means a cleaner handling of the composition which is increasingly important the higher the intended voltage is of the cable.

It is possible to use existing vulcanising tubes for the crosslinking of the insulating composition. The only modification that may be required is to provide the vulcanising tube with an inlet for steam. The possibility of using existing vulcanising tubes is an important advantage of the invention and makes it possible for a cable manufacturer to change the production from peroxide crosslinked cables to moisture cured silane group containing cables with no or only a minor investment.

The crosslinking temperature is lower at the present invention than at peroxide crosslinking. Thus, while the temperature in a vulcanising tube is about 400°C at peroxide crosslinking, as mentioned above, the temperature in a vulcanising tube used for crosslinking of the silane group containing polymer composition of the present invention is preferably only about 170-190°C. This lower crosslinking temperature means that less energy is consumed for the crosslinking at the present invention.

Compared to peroxide crosslinking the cost of the crosslinking operation is about the same or may even be lower at the present invention.

Compared to crosslinking using conventional prior art silanol condensation catalysts, such as DBTL as well as compared to crosslinking with peroxides, the present invention provides a much higher crosslinking speed.

Further, it is possible to carry out the crosslinking at a much lower temperature, such as room temperature, than with conventional silanol condensation catalysts (DBTL).

A particular advantage is that it is possible to use hydrolysable silane containing polymers for the insulating layer of medium to high voltage power cables of more

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than 20 kV, i.e. power cables with an insulating layer that is more than 5 mm thick.

A further important advantage is that the water treeing characteristics of the insulating layer are very good and much better than e.g. for peroxide crosslinked insulating layers. This is quite surprising because the specific sulphonic acid catalyst of formula I used in the present invention is an ionic compound and ionic compounds are known to adversely affect the water treeing characteristics. So far it has not been able to explain the reason for the excellent water treeing characteristics obtained with the present invention.

Having thus explained the present invention and its advantages it will now be illustrated by way of some non-limiting examples. All parts and percentages in the examples refer to weight, unless otherwise stated.

#### Example 1

This example illustrates the excellent water-tree retarding (WTR) characteristics obtained with the insulating composition of the present invention.

A composition according to the present invention was made into a cable insulation and its dielectric strength evaluated according to the so-called Model Cable Test developed by Alcatel AG & Co, Hannover, Germany, and described in an article by Land H.G., Schädlich Hans, "Model Cable Test for Evaluating the Ageing behaviour under Water Influence of Compounds for Medium Voltage Cables", Conference Proceedings of Jicable 91, 24-28 June 1991, Versailles, France. As a value of the dielectric strength is stated 63% of  $E_{max}$  from Weibull diagram in kV/mm. The dielectric strength was measured after ageing for 1000 hrs at 9 kV/mm in 85°C/70°C water.

The insulating composition of the present invention consisted of an ethylene copolymer base resin containing 2% by weight of vinyl trimethoxy silane with an MFR<sub>2</sub> of 1 g/10 min and a density of 0.923 g/cm<sup>3</sup>. The composition was compounded with a master batch consisting of 88.7% by

weight of ethylene/butyl acrylate copolymer (17% by weight of butyl acrylate;  $MFR_2 = 8$  g/10 min), 5% by weight of stabilizers and 1% of lubricant, 4% by weight of SI-116 (HDTMS; hexadecyl trimethoxysilane), 0.3% by weight of Ufacid K (DBSA; dodecylbenzene sulphonic acid), and 1% by weight of isododecane.

After crosslinking of the model cable according to the invention (referred to as Cable 1 below) in a water bath at 70°C for 72 hrs. Cable 1 was tested according to the Model Cable Test indicated above.

According to this test the insulating composition of Cable 1 had a breakdown strength of  $E_{max} = 79.6$  kV/mm at  $T = 0$  and  $E_{max} = 75.7$  kV/mm at  $T = 1000$  hrs.

The corresponding values for a reference model cable (referred to as Reference Cable 1 below) where the insulating composition consisted of a standard LDPE with  $MFR_2 = 1.9$  g/10 min and a density =  $0.9225$  g/cm<sup>3</sup> and crosslinked with 2% by weight of peroxide crosslinking agent was  $E_{max} > 89$  kV/mm at  $T = 0$  and  $E_{max} = 34.3$  kV/mm at  $T = 1000$  hrs.

#### Example 2

Example 1 was repeated with the exception that the master batch of the insulating composition according to the invention contained 0.7% by weight of the Ufacid K (DBSA) and 88.3% by weight of the ethylene butylacrylate copolymer. The model cable made with this composition as the insulating layer is referred to as Cable 2.

The results of the model cable test were  $E_{max} > 81.7$  kV/mm at  $T = 0$  and  $E_{max} = 54.4$  kV/mm at  $T = 1000$  hrs.

The Cable 2 according to the invention and the Reference cable 1 of Example 1 were evaluated for water treeing. The results are given in Table 1.



TABLE 1

	Bow-tie trees No./mm <sup>3</sup>	Bow-tie trees Longest (μm)	Vented trees No./mm <sup>3</sup>	Vented trees Longest (μm)
Cable 2	21	110	0.04	90
Ref. cable 1	460	511	0.34	1070

It is evident from both Example 1 and Example 2 that the insulating composition according to the present invention gives superior WTR performance as an insulating layer of a medium to high voltage power cable compared to a standard peroxide crosslinked cable.

#### Example 3

In order to show the improved crosslinking rate of the insulating composition of the present invention compared to an insulating composition using a conventional silanol condensation catalyst (DBTL) the following test was made.

Cables were made having a 1.5 mm<sup>2</sup> conductor surrounded by a 0.7 mm thick insulating layer.

The insulating layer of the cable according to the present invention (referred to as Cable 3) consisted of the same base resin as the insulating layer of Cable 1 in Example 1 compounded with 5% by weight of a master batch consisting of 87.3% by weight of ethylene butyl acrylate copolymer (17% by weight of butyl acrylate; MFR<sub>2</sub> = 8 g/10 min), 5% by weight of stabilizers and 1% of lubricant, 4% by weight of SI-116 (HDTMS), 1.7% by weight of Ufacid K, and 1% by weight of isododecane.

The insulating layer of the reference cable (referred to as Reference cable 3) consisted of the same base resin, but compounded with 5% by weight of a master batch consisting of 57.5% by weight of ethylene butyl acrylate copolymer (27% by weight of butyl acrylate), 34% by weight of LDPE, 4.5% by weight of stabilisers, 1% by

weight of zinc stearate, and 3% by weight of DBTL. The master batch had an  $MFR_2 = 5$  g/10 min.

The crosslinking rate was determined according to IEC-811-2-1-9 (hot set method) by measuring the thermal deformation at 200°C and a load of 20 N/cm<sup>2</sup> after various times of crosslinking at 23°C and 50% R.H. The percentage hot set elongation for Cable 3 with the insulating composition according to the invention was about 45, 38, and 28% after 2, 3, and 4 days of crosslinking, respectively. The percentage hot set elongation for Reference cable 3 was about 148, 102, 77 and 52% after 2, 3, 4, and 7 days of crosslinking, respectively. Although the cables tested in this example are not medium to high voltage power cables, it is nevertheless evident that the insulating composition used at the present invention crosslinks about four times faster than a conventional insulating composition.

#### Example 4

The crosslinking of a medium to high voltage power cable with an insulating layer according to the present invention was tested and compared to the crosslinking of a similar cable, but with an insulating layer with a conventional DBTL silanol condensation catalyst.

The cable according to the present invention had an insulating layer with the same composition as that of Cable 3 in Example 3.

The reference cable (referred to as Reference Cable 4) had an insulating layer with the same base resin as that of Cable 4 compounded with 5% by weight of a master batch consisting of 57% by weight of ethylene butyl acrylate copolymer (27% by weight of butyl acrylate), 34% by weight of LDPE, 5.7% by weight of stabilisers, 1% by weight of zinc stearate, and 2% by weight of DBTL. The master batch had an  $MFR_2 = 5$  g/10 min.

The insulating layers of Cable 4 and Reference cable 4 were each 9 mm thick (30 kV cables).

Cable 4 and Reference cable 4 were crosslinked in a 90°C water bath for different, predetermined time periods. After each time period a 1.2 mm thick sample was taken 2.7 mm from the outer surface of the cable and the crosslinking was evaluated by the hot set method according to IEC-811-2-1-9 mentioned above.

Cable 4 according to the present invention had a hot set elongation of 85, 75, and 45% after 3.5, 4, and 4.5 hrs of crosslinking respectively, while Reference cable 4 had a hot set elongation of 205, 155, 130, 115, 105, 70, and 65% after 4, 4.5, 5, 5.5, 6, 7, and 8 hrs of crosslinking, respectively.

It is evident that crosslinking of medium to high voltage power cable insulating layers according to the present invention is very quick and effective compared to the crosslinking of a similar insulating layer with a conventional DBTL catalyst, and that the medium to high voltage power cable insulating composition presents a feasible alternative to conventional peroxide crosslinked insulating layers of medium to high voltage power cables.

#### Example 5

Another evaluation of the crosslinking rate of the insulating layer composition according to the present invention was made in this example.

Two power cables with different insulating layers according to the present invention were made. The first cable (referred to as Cable 5a) had an insulating layer of the same composition as Cable 3 in Example 3. The second cable (referred to as Cable 5b) had a hydrophilic base resin consisting of a terpolymer of ethylene, 17% by weight of butyl acrylate, and 2% by weight of VTMS (vinyl trimethoxysilane) compounded with 5% by weight of the same master batch as Cable 5a.

The reference cable (referred to as Reference cable 5) had an insulating layer including the same base resin as Cable 5b compounded with the same master batch as that of Reference cable 3 of Example 3.

The insulating layers of Cables 5a, 5b, and Reference cable 5 each had a thickness of 5.5 mm (20 kV cables).

Cables 5a, 5b and Reference cable 5 were crosslinked in a 90°C water bath during different, predetermined time periods. After each time period a 1.2 mm thick sample was taken 2.9 mm from the outer surface of the cable and the crosslinking was evaluated by the hot set method according to IEC-811-2-1-9 mentioned above.

Cable 5a according to the present invention failed the hot set test after 8 hrs of crosslinking, and had a hot set elongation of 45 and 35% after 12 and 17 hrs crosslinking respectively. Cable 5b according to the present invention failed the hot set test after 2 hrs of crosslinking and had a hot set elongation of 20, 15, 15 and 15% after 4, 8, 12, and 17 hrs crosslinking respectively. Reference cable 5 failed the hot set test after 17 hrs of crosslinking.

Again, this example illustrates the feasibility of the composition of the present invention as an excellent insulating layer for a medium to high voltage power cable and as a replacement for conventional insulating layers of such cables. It also illustrates the increased crosslinking rate obtained when the composition of the insulating layer is made hydrophilic.

#### Example 6

In order to show the possibility of crosslinking a power cable according to the present invention with the use of vulcanising tube and the improved crosslinking rate of the insulating composition of the present invention compared to a conventional silanol condensation catalyst (DBTL) the following tests were made.

Three cables were made having a 50 mm<sup>2</sup> (8.05 mm diameter) aluminium conductor surrounded by a 0.5 mm thick semiconducting layer and then a 6.4 mm thick insulating layer. The cables did not have any outer semiconducting layer.

The composition of the insulating layer of the cables was varied. The first cable which was a cable according to the present invention (referred to as Cable 6a) had a composition corresponding to that of Cable 3 of Example 3 above (a silane group containing ethylene copolymer). The second cable which was also a cable according to the present invention (referred to as Cable 6b) had an insulating layer composition corresponding to that of Cable 5b of Example 5 above (a hydrophilic silane group containing ethylene/butyl acrylate terpolymer). The third cable which was not according to the present invention (referred to as Reference cable 6) had a composition corresponding to that of Reference cable 3 of Example 3 above (DBTL as catalyst).

After extrusion of the cables they were crosslinked in a 20 m long laboratory vulcanising tube with saturated steam at a pressure of 1.0 MPa gauge. The vulcanising tube had four heating zones with the temperatures set at 179, 178, 178 and 135°C for Cable 6a; 177, 179, 181, and 183°C for Cable 6b; and 181, 179, 181 and 184°C for Reference cable 6. After the heating zones followed a cooling zone with cooling water. The line speed was 2 m/min for all three cables.

The crosslinking was evaluated by measuring the Hot Set at 200°C and a force of 20 N/cm<sup>2</sup> of a cut of thickness 1.2 mm taken at a distance of between 2.9 and 4.1 mm from the outer surface. The obtained values appear from Table 2:

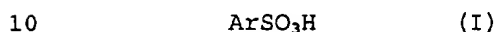
TABLE 2

Elongation in	Directly after CV	After	After	After
%	tube	1 day	4 days	6 days
Cable 6a	60	45	-	-
Cable 6b	50	30	-	-
Ref.cable 6	broke	broke	100	70

The crosslinking was evaluated as Hot Set for cables 6a, 6b and Reference cable 6 according to IEC-811-2-1-9 (hot set method) by measuring the thermal deformation at 200°C and a load of 20 N/cm<sup>2</sup> after various times of crosslinking at 23°C and 50% RH. Samples of 1.2 mm thickness were taken at a distance of between 2.9 and 4.1 mm from the outer surface of the insulating layer of the cables. The obtained values appear from Table 2. The percentage hot set elongation for Cable 6a was 60% and 45% after 0 days (directly after the exit from the vulcanising tube) and 1 day, respectively. The percentage hot set elongation for Cable 6b was 50%/0 days and 30%/1 day. The percentage hot set elongation for Reference cable 6 was: - (breakage)/0 days; - (breakage)/1 day; 100%/4 days; and 70%/6 days. The superiority of Cable 6a and, in particular Cable 6b of the invention over Reference cable 6 with a conventional DBTL catalyst is clearly evident.

## CLAIMS

1. A composition for an insulating layer of a medium to high voltage power cable, characterised in that the composition comprises a crosslinkable polymer with hydrolysable silane groups, and a silanol condensation catalyst of formula I



or a precursor thereof, Ar being a benzene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical(s) is 8-20, or a naphthalene ring substituted with at least one hydrocarbyl radical such that the total number of carbon atoms of the hydrocarbyl radical(s) is 4-18, and the catalyst of formula I containing 14-28 carbon atoms in total.

2. A composition as claimed in claim 1, wherein the composition is hydrophilic.

3. A composition as claimed in claim 2, wherein the crosslinkable polymer has hydrophilic groups selected from siloxane, amide, anhydride, carboxylic, carbonyl, hydroxyl, and ester groups.

4. A composition as claimed in any one of the preceding claims, wherein the crystalline part of the polymer is at most 60% by weight.

5. A composition as claimed in any one of the preceding claims, wherein the hydrocarbyl radical in formula I is an alkyl substituent with 10-18 carbon atoms.

6. A composition as claimed in claim 5, wherein the alkyl substituent has 12 carbon atoms and is selected from dodecyl and tetrapropyl.

7. A composition as claimed in any one of the preceding claims, wherein the polymer composition includes 0.0001-3% by weight of silanol condensation catalyst.

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8. A medium to high voltage power cable comprising a conductor surrounded in order by an inner semi-conducting layer, an insulating layer, and an outer semi-conducting layer, c h a r a c t e r i s e d in that the insulating layer has a thickness of more than 2 mm and comprises the crosslinked product of a composition according to any one of claims 1-8.

9. A medium to high voltage power cable as claimed in claim 8, wherein the insulating layer has a thickness of more than 5 mm.

10. A process of preparing a medium to high voltage power cable according to claim 8 or 9, in which a conductor is surrounded in order by an inner semi-conducting layer, an insulating layer comprising a crosslinkable polymer with hydrolysable silane groups, and an outer semi-conducting layer to form a cable, c h a r a c t e r i s e d in that the cable is crosslinked in the presence of steam at a superatmospheric pressure.

11. A process according to claim 10, wherein the crosslinking is carried out in a vulcanising tube.

12. A process according to claim 10 or 11, wherein the crosslinking is carried out at a pressure of 0.2-2.5 MPa.

13. A process according to claim 12, wherein the crosslinking is carried out at a pressure of 0.8-1.2 MPa.

14. A process according to any one of claims 10-13, wherein the crosslinking is carried out in the presence of saturated steam.



CLAIMS

1. A composition for an insulating layer of a medium  
5 to high voltage power cable, c h a r a c t e r i s e d  
in that the composition comprises a crosslinkable polymer  
with hydrolysable silane groups, and a silanol condensa-  
tion catalyst of formula I

10  $\text{ArSO}_3\text{H}$  (I)

or a precursor thereof, Ar being a benzene ring substi-  
tuted with at least one hydrocarbyl radical such that the  
total number of carbon atoms of the hydrocarbyl  
15 radical(s) is 8-20, or a naphthalene ring substituted  
with at least one hydrocarbyl radical such that the total  
number of carbon atoms of the hydrocarbyl radical(s) is  
4-18, and the catalyst of formula I containing 14-28  
carbon atoms in total.

20 2. A composition as claimed in claim 1, wherein the  
composition is hydrophilic.

3. A composition as claimed in claim 2, wherein the  
crosslinkable polymer has hydrophilic groups selected  
from siloxane, amide, anhydride, carboxylic, carbonyl,  
25 hydroxyl, and ester groups.

4. A composition as claimed in any one of the pre-  
ceding claims, wherein the crystalline part of the  
polymer is at most 60% by weight.

5. A composition as claimed in any one of the pre-  
ceding claims, wherein the hydrocarbyl radical in formula  
30 I is an alkyl substituent with 10-18 carbon atoms.

6. A composition as claimed in claim 5, wherein the  
alkyl substituent has 12 carbon atoms and is selected  
from dodecyl and tetrapropyl.

35 7. A composition as claimed in any one of the pre-  
ceding claims, wherein the polymer composition includes  
0.0001-3% by weight of silanol condensation catalyst.

8. A medium to high voltage power cable comprising a conductor surrounded in order by an inner semi-conducting layer, an insulating layer, and an outer semi-conducting layer, c h a r a c t e r i s e d in that the insulating  
5 layer has a thickness of more than 2 mm and comprises the crosslinked product of a composition according to any one of claims 1-8.

9. A medium to high voltage power cable as claimed in claim 8, wherein the insulating layer has a thickness  
10 of more than 5 mm.

10. A process of preparing a medium to high voltage power cable according to claim 8 or 9, in which a conductor is surrounded in order by an inner semi-conducting layer, an insulating layer comprising a crosslinkable  
15 polymer with hydrolysable silane groups, and an outer semi-conducting layer to form a cable, c h a r a c - t e r i s e d in that the cable is crosslinked in the presence of steam at a superatmospheric pressure.

11. A process according to claim 10, wherein the  
20 crosslinking is carried out in a vulcanising tube.

12. A process according to claim 10 or 11, wherein the crosslinking is carried out at a pressure of 0.2-2.5 MPa.

13. A process according to claim 12, wherein the  
25 crosslinking is carried out at a pressure of 0.8-1.2 MPa.

14. A process according to any one of claims 10-13, wherein the crosslinking is carried out in the presence of saturated steam.